CERAMIC RELIABILITY FOR MICROTURBINE HOT-SECTION COMPONENTS

Reliability Evaluation of Microturbine Components

H. T. Lin, M. K. Ferber, T. P. Kirkland, and P. F. Becher
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, TN 37831-6068
Phone: (865) 576-8857, E-mail: linh@ornl.gov

Objective

Evaluate and document the long-term mechanical properties of very small specimens machined from ceramic components (e.g., blades, nozzles, vanes, and rotors) in as processed and after engine testing at ambient and elevated temperatures under various controlled environments. This work will allow microturbine companies to verify mechanical properties of components and apply the generated database in advanced design and lifetime prediction methodologies. The work also provides a critical insight into how the microturbine environments influence the microstructure and chemistry, thus mechanical performance, of materials.

Highlights

A US-Japan joint research project has been established between Oak Ridge National Laboratory (ORNL) and Synergy Materials Research Center (SMRC), Nagoya, Japan under US-Japan Science and Technology Cooperation on Climate Change. The objectives of this joint project are to develop (1) the next generation of self-reinforced silicon nitride and (2) environmental barrier coating (EBC) for the land base gas turbine applications. The silicon nitride ceramics fabricated via the beta-Si_3N_4 seeds and tape-casting process with and without EBC will be developed by SMRC. ORNL will be responsible for evaluation of the effect of long-term steam exposure on the chemical stability and thus mechanical reliability of the material system.

Technical Progress

Studies of mechanical properties of samples extracted from SN282 silicon nitride nozzles after 100 h engine test at Solar Turbines, Inc., were carried out during this reporting period. The biaxial flexure strength was measured using the ball-on-ring arrangement, as shown in Figure 1. Specimens were machined from both the suction (convex) and pressure (concave) side of airfoil surfaces by first diamond core drilling small cylinders having nominal diameters of 5.5 mm. Each cylinder was then machined on one face only until the thickness was 0.4 to 0.5 mm. In this way, one face of each specimen always consisted of the exposed surface of the airfoil. During testing, this exposed surface was loaded in tension. The data reported were the average value of at least 5 test specimens. The mechanical results showed that the 100-h samples exhibited similar strength as compared with those obtained from the as-received nozzles, as shown in Fig. 2. Also, the obtained strength data are not strongly dependent upon the airfoil location from which the samples were extracted. The results suggest that the 100-h engine test has little influence on the mechanical strength of SN282 silicon nitride nozzles, consistent with the mechanical results obtained from SN282 vanes after engine tests up to 1818 h by Rolls-Royce Allison. Nonetheless, the samples with as-processed surface still exhibited a strength value, which was ~ 46% lower than those machined from shroud region with machined surface. The much lower strength obtained for the samples from airfoil regions with as-processed surface was attributed to the presence of processing flaws, i.e., pores.

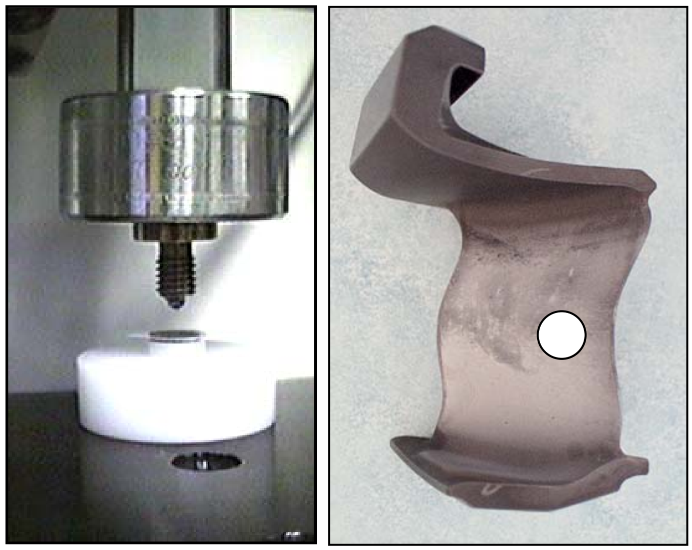


Figure 1. Biaxial testing system and samples extracted from airfoil of nozzle.

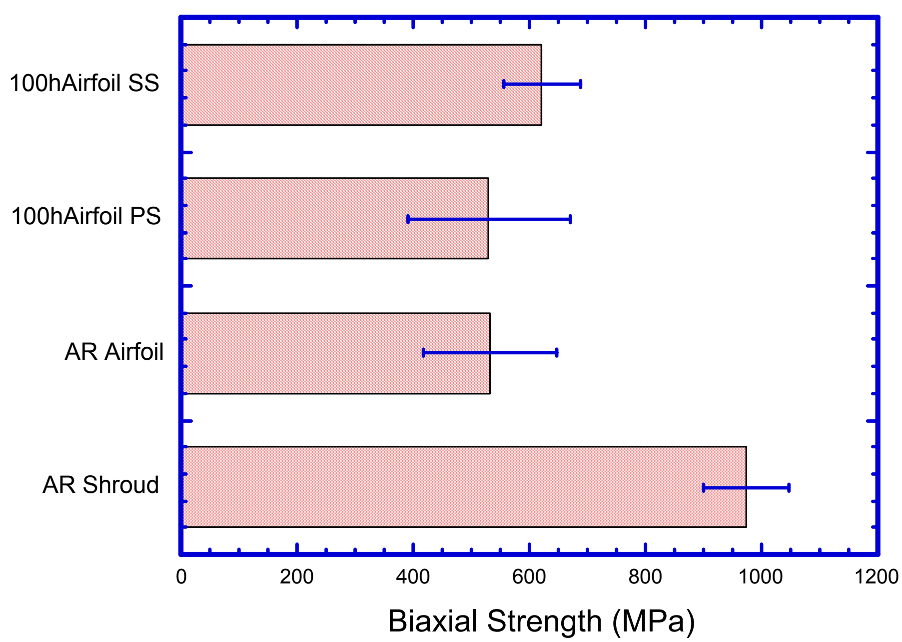
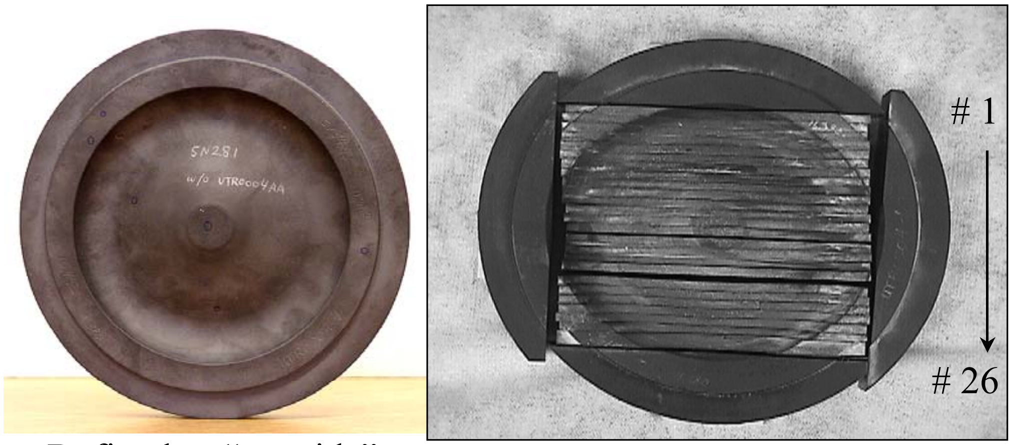


Figure 2. Biaxial strength of samples extracted from airfoil of SN282 nozzle after 100h engine test.



Defined as “top side”

Figure 3. Photos show (a) the SN281 microturbine rotor blank and (b) sample slices machined from rotor.

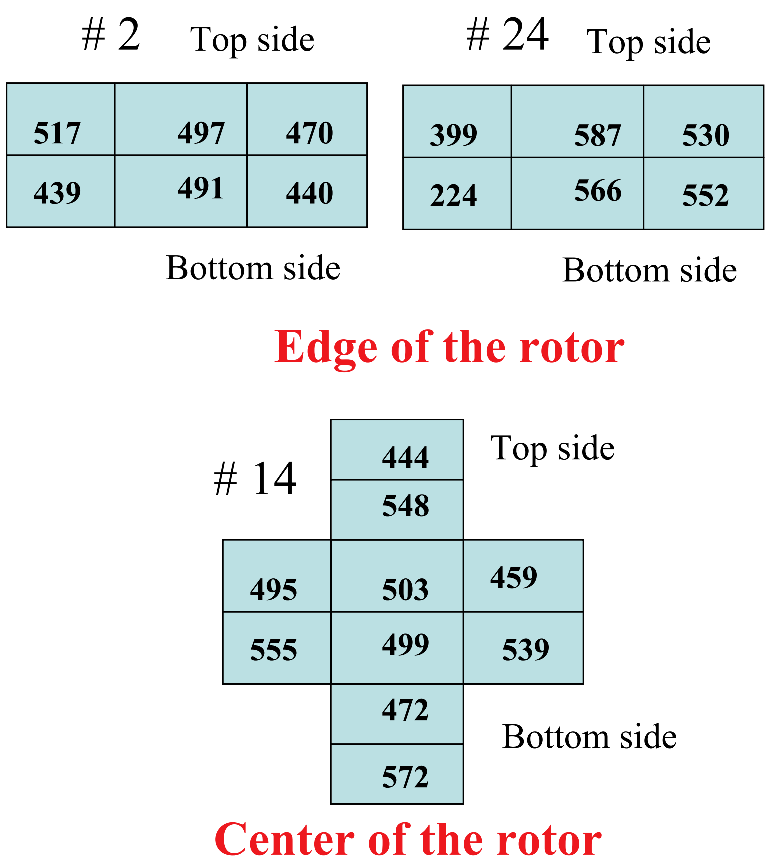


Figure 4. Flexural strength distribution at 1204°C at 30 MPa/s in air as a function of location in SN281 microturbine rotor blank.

Evaluation of the inert characteristic strength at 1204°C for bend bars machined from UTRC SN281 microturbine rotor blank has been completed during this reporting period. The test results at room temperature have been reported previously. Figure 3 shows the specific locations where the test specimens were machined from the rotor blank. Mechanical results at 1204°C showed that the measured strength varied from location to location, as shown in Fig. 4, which was similar to those obtained at room temperature. Also, the strength differences for bend bars machined from the same slice could vary over 100%, as seen in the material from the edge of rotor (designated No. 24). Note that no strength degradation was observed when tested at 1204°C. Fractography analysis indicated that the strength limiting flaw for bend bars with very low strength is mainly due to the processing pores with size as large as 0.5mm, similar to those reported previously for the room-temperature tested specimens.

Status of Milestones

1. Complete characterization of microstructure and mechanical properties for Ingersoll-Rand SN237 silicon nitride microturbine rotors by September 2003. On schedule.

Industry Interactions

Communication with John Holowczak at UTRC to discuss the mechanical results at 1204°C for Kyocera SN281 ST5 rotor blank.

Two Kyocera SN281 microturbine rotors were received from John Holowczak at UTR.

Communication with Josh Kimmel and Mark van Roode at Solar Turbines on the biaxial strength results obtained for SN282 silicon nitride nozzles after 100h engine test.

The first set of Norton NT154 billets was received from Vimal Pujari at Saint-Gobain for mechanical strength as well as steam exposure evaluations.

Communication with Jim Kesseli at Ingersoll-Rand Energy Systems to discuss the delivery date of Kyocera SN237 microturbine rotors.

Communication with Russ Yeckley at Kennemetal about the delivery of the first set of α/β SiAlON materials for mechanical property evaluation.

Problems Encountered

None

Publications/Presentations

1. H. T. Lin, M. K. Ferber, and T. P. Kirkland, "Evaluation of Mechanical Stability of a Commercial SN88 Silicon Nitride at Intermediate Temperatures," accepted for publication in *J. Am. Ceram. Soc.*

Long-Term Testing in Water Vapor Environments

M. K. Ferber and H. -T. Lin
Metals and Ceramics Division
Oak Ridge National Laboratory
P.O. Box 2008, Oak Ridge, TN 37831-6068
Phone: (865) 576-0818, E-mail: ferbermk@ornl.gov

Objective

The objective of this project is to develop test facilities for evaluating the influence of high-pressure and high-temperature water vapor upon the long-term mechanical behavior of monolithic ceramics having environmental barrier coatings.

Highlights

The steam injection system was also used to evaluate an environmental barrier coating (EBC) applied to a silicon carbide flexure specimen.

Technical Progress

The effort this period continued to focus on the evaluation of the effects of water vapor on the creep behavior of silicon nitride ceramics utilizing the steam injection system (Figure 1) described in the previous quarterly report. Figure 2 illustrates the surface recession of three tensile specimens exposed at 1200 and 1288°C for various periods of time. The measured recession rates, R, were normalized with respect to velocity and pressure using the modified NASA-Glenn expression:

$$R\ (\mu m/h)/[v^{1/2}\ (P_{H2O})^2\ (P_{total})^{-1/2}] = \exp(-E/RT) \qquad (1)$$

where E is the activation energy, T is the absolute temperature (in K), v is the gas stream velocity (in m/s), P_{H2O} is the pressure (in atm) of the water vapor, and P_{total} is the total pressure (in atm). As indicated in Figure 3, the recession results obtained in the present study compared well with similar data reported in the literature [1-5].

The steam injection system was also used to evaluate an environmental barrier coating (EBC) applied to a silicon carbide flexure specimen (Figure 4) and exposed for 260 h at 1200°C. Because the diameter of injection tube was slightly larger than the width of the specimen, the steam was able to flow over the uncoated sides of the flexure specimen. Consequently these uncoated surfaces did experience significant recession (Figure 5). However, subsequent examination of polished cross-sections representing the center of the injection region showed that the EBC was quite effective in reducing the oxidation of the SiC substrate.

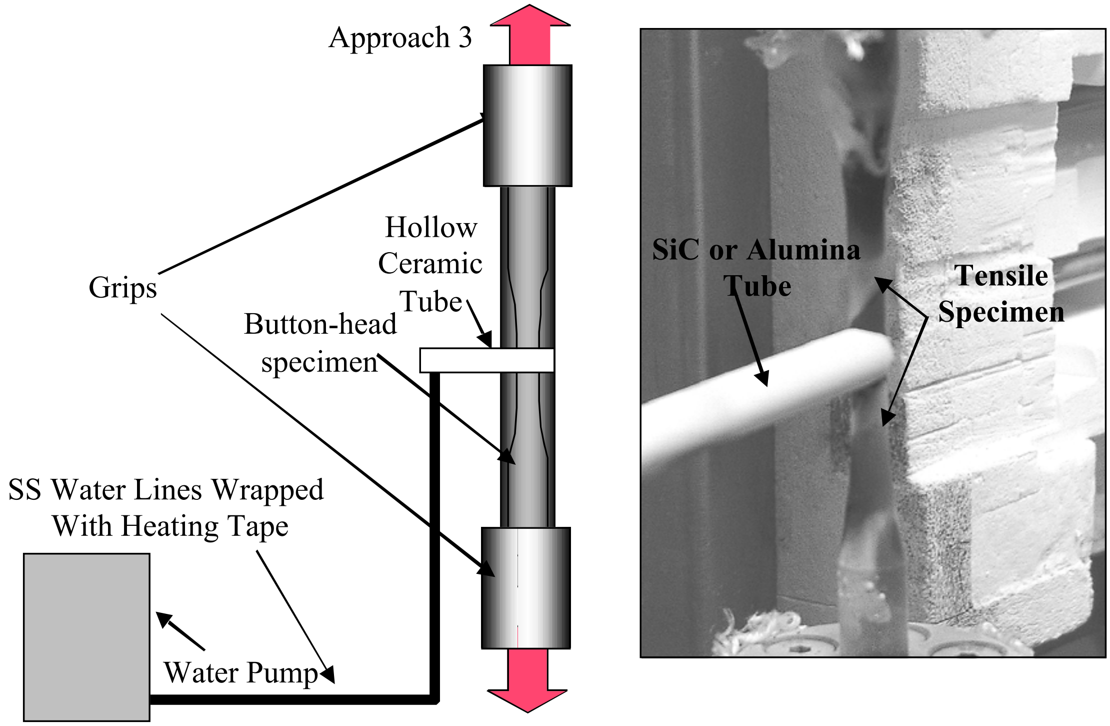
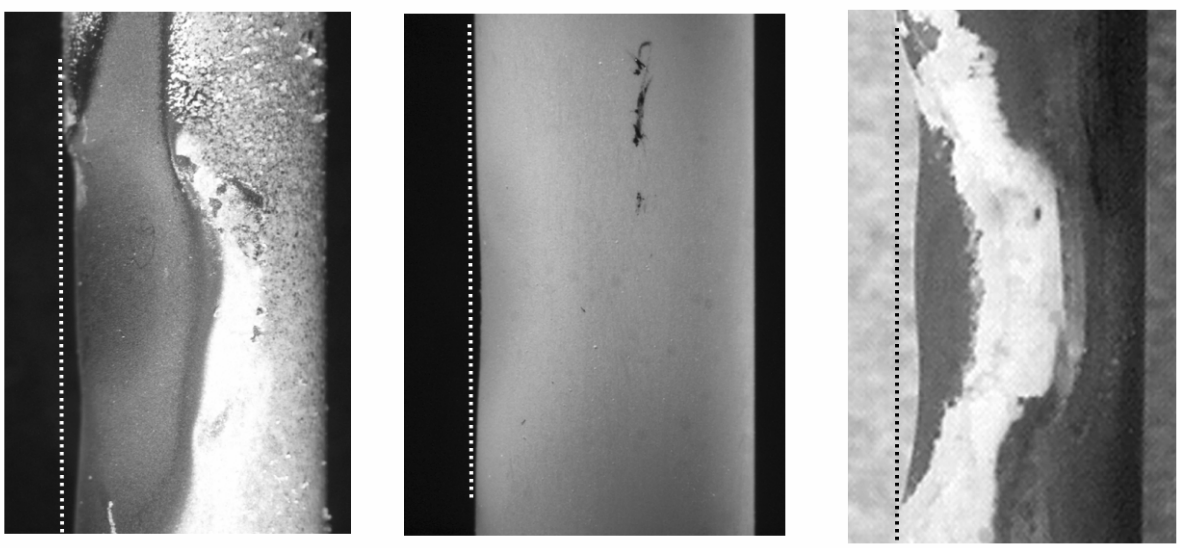


Figure 1: Direct Steam Injection System.



SA SiC- 291 1200°C & 500 h with Water Vapor (250 μm recession)

NT154-1 1200°C & 500 h with Water Vapor (100 μm recession)

NT164-81 1288°C, 150 MPa, & 2000 h with Water Vapor (500 μm recession)

Figure 2. Side view of several tensile specimens evaluated with the steam injection system. In all three cases the steam flow was from left to right.

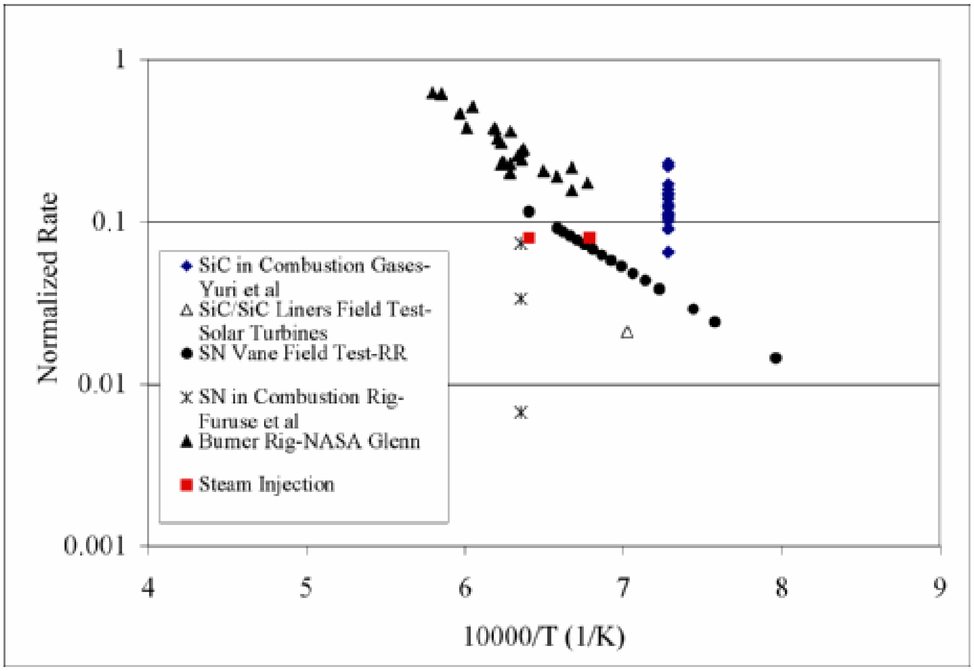


Figure 3. Comparison of normalized recession rate versus 1/T data obtained in present study with similar data reported in the literature.

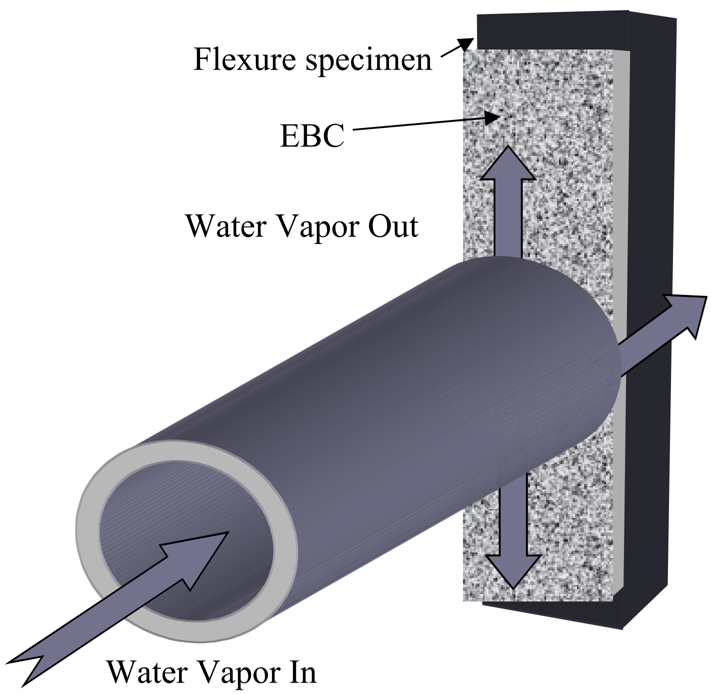


Figure 4. Arrangement used to evaluate the stability of an EBC on a silicon carbide flexure specimen subjected to a steam environment. The flexure bar was mounted to the gage section of a buttonhead tensile specimen.

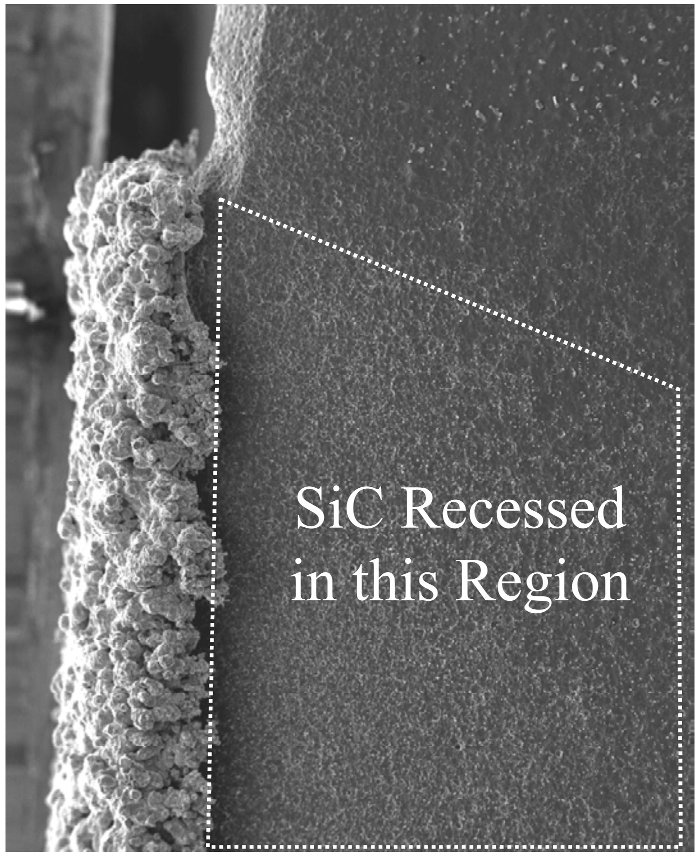


Figure 5. Side view of SiC/EBC specimen exposed to steam for 260 h at 1200°C.

Status of Milestones

Milestone 1: Design, fabricate, and evaluate steam containment system for existing creep-stress rupture rigs and issue letter report (April 1, 2001/Delayed 12 months/Completed April 10, 2002).

Milestone 2: Conduct preliminary environmental stability tests on uncoated SN282 and issue letter report (July, 2002/ Delayed 4 months).

Milestone 3: Modify 4 test frames to accommodate direct steam injection system (March 2002/Completed June 2002).

Industry Interactions

During the 27th Annual Cocoa Beach Conference and Exposition on Advanced Ceramics & Composites discussions were held with (a) Jim Kesselli of Ingersoll-Rand Energy Systems concerning characterization of the ceramic rotors to be used in their microturbine demonstration project, (b) Russ Yeckley of Kennametal concerning delivery of specimens for mechanical and Keiser rig testing, and (c) Vimal Pujari of Saint-Gobain concerning testing of their first set of specimens.

Problems Encountered

None

Publications

None

References

[1] Y. Furuse, T. Teramae, T. Tsuchiya, F. Maeda, Y. Tsukuda, and K. Wada, "Application of Ceramics to a Power Generating Gas Turbine" pp in Ceramic Gas Turbine Design and Test Experience: Progress in Ceramic Gas Turbine Development, Vol. 1, edited by Mark van Roode, Mattison K. Ferber, and David W. Richerson, ASME International, 2002.

[2] I. Yuri, T. Hisamatsu, Y. Etori and T. Yamamoto, "Degradation of Silicon Carbide in Combustion Gas Flow at High-Temperature and Speed," 2000-GT-0665, 2000.

[3] J. L. Smialek, R. C. Robinson, E. J. Opila, D. S. Fox, and N. Jacobson, "SiC and Si_3N_4 Recession due to SiO_2 Scale Volatility Under Combustor Conditions, *" Advanced Composite Materials*, Vol. 8, No. 1, pp. 33-45, (1999).

[4] M. K. Ferber, H. T. Lin, V. Parthasarathy, and R. A. Wenglarz, "Degradation of Silicon Nitrides In High Pressure, Moisture Rich Environments," ASME paper 2000-GT-661, presented at the ASME TURBO EXPO LAND, SEA & AIR, Munich, Germany, 8-11 May 2000.

[5] H. T. Lin, M. K. Ferber, W. Westphal, and F. Macri, "Evaluation of Mechanical Reliability of Silicon Nitride Vanes After Field Tests in an Industrial Gas Turbine," ASME 2002-GT-30629, presented and published in the Proceedings of at TURBO EXPO Land Sea, and Air 2002, June 3-6, Amsterdam, The Netherlands.

NDE Technology Development for Microturbines

W. A. Ellingson, J. G. Sun, E. R. Koehl, Z. Metzger, and C. Deemer
Argonne National Laboratory
9700 South Cass Avenue, Argonne, IL 60439
Phone: (630) 252-5058, E-mail: Ellingson@anl.gov

Objective

The objective of this project is development of nondestructive evaluation/characterization (NDE/C) technologies for: (1) evaluating low-cost monolithic ceramics for hot section components of microturbines or industrial gas turbines, (2) evaluating environmental barrier coatings (EBCs) for monolithic ceramics and ceramic matrix composites, and (3) evaluating other materials which are part of the technology to advance DER technologies such as ceramic-metal joints. The project is directly coupled to other Office of Distributed Energy and Electrical Reliability projects focused on materials developments directed towards low-cost, high volume monolithic ceramics, environmental barrier coating systems and related technologies such as ceramic-metal joining.

Highlights

There are three highlights this period. First, we have made significant improvements in the necessary digital imaging correction factors for the new large area flat panel detector that has a CsI scintillator. This is important for 3D x-ray imaging of the monolithic parts. Second, we completed the transfer of a 420KVp x-ray head and x-ray imaging system from Oak Ridge national Lab to Argonne and third, have made additional progress regarding testing of new EBCs.

Technical progress

Technical work this period focused on 2 areas: (1) further developments towards volumetric, 3D, X-ray imaging for improving the reliability and processing methods of low-cost monolithic ceramic materials, and (2) work on oxide-based ceramic composites.

The technical progress to be reported will focus on: a)- the large-area flat panel detectors, b)- dismantling, moving and re-installing the 420 KVp x-ray head and c)- additional results on the oxide-based composites.

<u>NDE development for on-line low-cost monolithics</u>

Work this period focused on two areas: (1) data acquisition tests using large-area X-ray detectors, and (2) installing a new 420 KVp x-ray head

<u>Large Flat Panel X-ray detector</u>

We have been discussing issues related to the new 40 cm by 40 cm CsI scintillator large-area flat panel x-ray detector with 200 µm square pixels. As opposed to the 400 um square pixel detector we have been using that uses LANEX-fast as a scintillator, this area detector has had major digital image distortions had been occurring. For whatever the reason, when an object is placed in front of the detector, even though a flat field correction had been implemented, there are so-called "ghost" type images that develop. When using such data sets in a tomographic image data sets, these result in severe ring artifacts in the reconstructed CT image data. Shown in Figs. 1 and 2 respectively are a corrected flat field image and an example of a previous image when a 12.5mm thick plate was placed in front of the detector. In order to properly correct these images, after extensive discussions with the manufacturer, in Wiesbaden, Germany, we implemented a correction factor that results in a projection image with the 12.5 mm thick plate as shown in Figure 3.

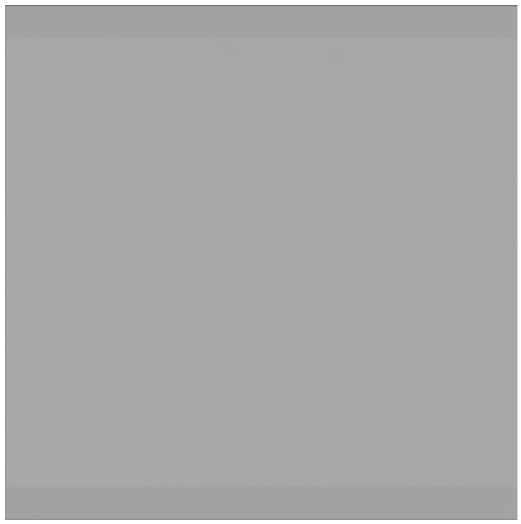


Fig. 1. Projection image on the CsI scintillator large area X-ray detector showing a desired flat screen.

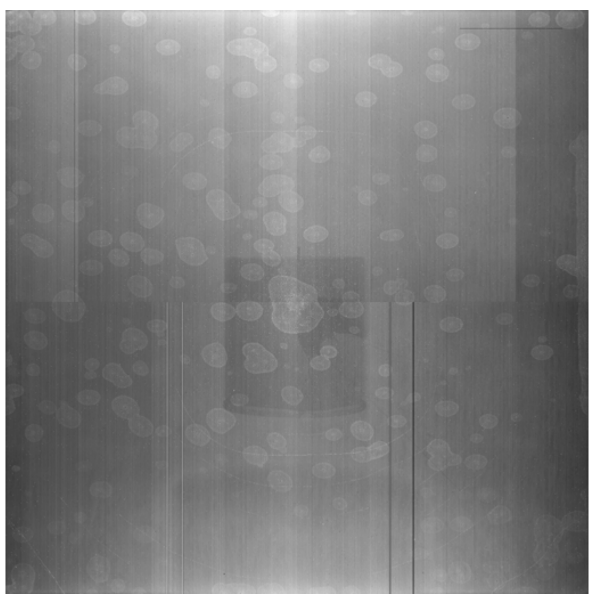


Fig. 2. Projection image on the CsI scintillator screen when a 12.5mm thick aluminum plate was placed in front of the screen with the old flat field correction and showing the "'mottling" pattern.

Fig. 3. Projection image on the CsI scintillator screen when a 12.5mm thick aluminum plate was placed in front of the screen with the new flat field correction. Note the “mottling” pattern is now gone.

Using this new correction, we obtained a sample data set using as a test sample a 44.5 mm diameter green-state ceramic cylinder shown schematically in Figure 4. To be noted is that this sample has been designed so that we can insert rods of 4.76 mm in diameters of different densities. This was scanned with the microfocus x-ray head and with the CsI RID1620 detector system with the following parameters. The x-ray head used 130KVp with 0.35mA and the detector was set at 1.625 seconds for integration time. The resulting 3D x-ray image was very good and one cross section is shown below in Figure 5.

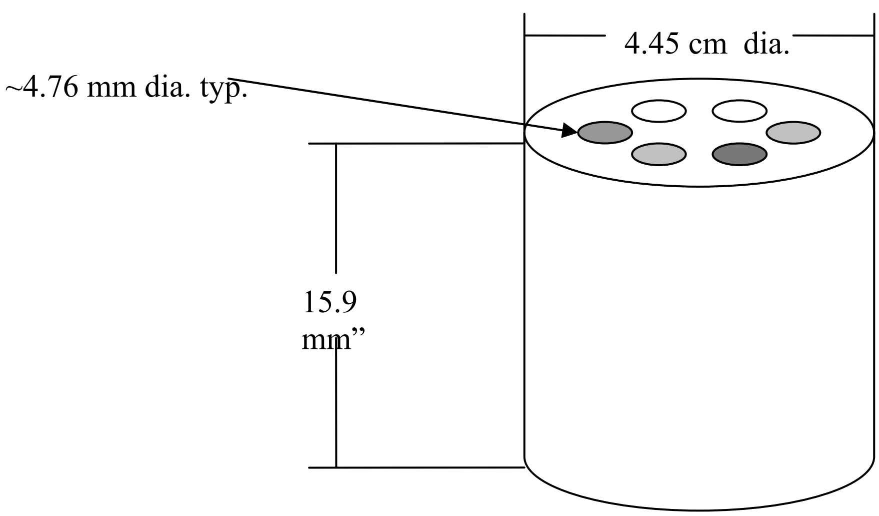


Figure 4. Schematic Diagram of 44.5 mm diameter cylinder used for CT study using new correction of the CsI large-area x-ray detector

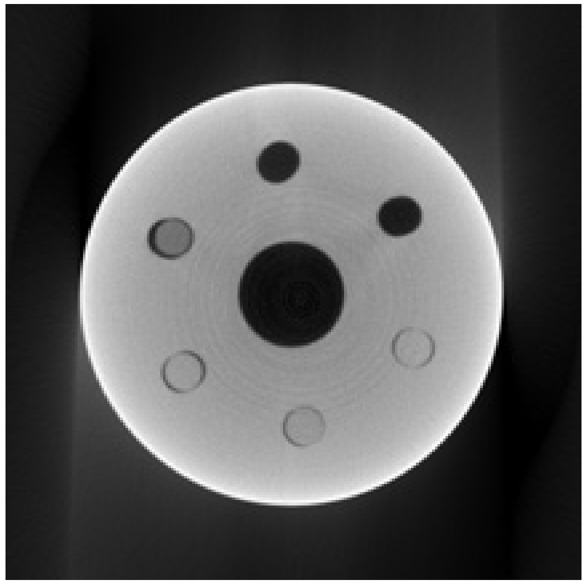


Figure 5. Single x-ray tomographic cross-section taken out of the 3D x-ray Data set obtained using the CsI large area detector but with the new flat field Correction. Data taken from sample shown schematically in Figure 4

Moving and re-installing 420KVp x-ray head

This period, after extensive discussions and completion of necessary transferring documentation, an available Phillips 420 KVp x-ray head and x-ray imaging system at Oak Ridge National Laboratory was transferred to Argonne. This capability is a necessary capability in order to allow the Argonne-developed 3D x-ray imaging technology to be implemented on the large ceramic components under development for the microturbine hot section components. Until this time, the Argonne capability was limited to a 320 KVp x-ray head and this simply was not sufficient for proper signal to noise ratios necessary for high defect definition in the microturbine components. The x-ray head was moved using a commercial carrier and is being installed in the x-ray imaging lab at Argonne. Next period, initial test results will be provided as all wiring and safety interlocks have been installed.

Oxide-based Composites

Work continued this period relative to development for oxide-based ceramic composites. This work is being done in conjunction with Dr. Dave Carruthers. NDE data were acquired on several samples that had been exposed in the ORNL Keiser rig. The samples previously had been examined after 1000 hours and these data were reported last period. This period we are reporting the data after 2000 hours. To be noted in the through-transmission x-ray images, Figure 6, is the development of significant apparent porosity as noted by the "black" regions distributed through-out the test section. The thermal diffusivity images, shown in Figure 7, do not seem to be so sensitive to theses apparent porosity changes. This thermal imaging technology for oxide/oxide composites is an area in need of development and is being addressed in a companion program.

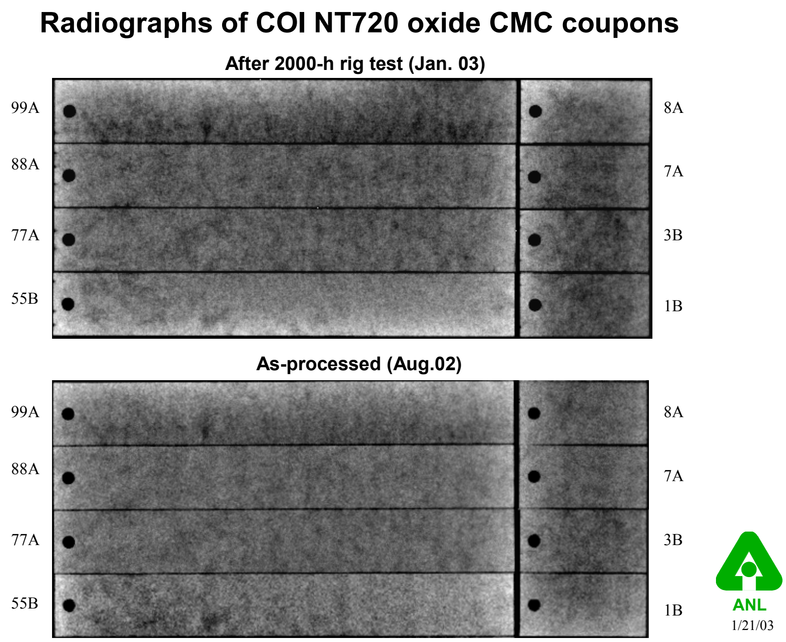


Fig. 6. Comparison of before and after through-transmission projection X-ray images of oxide-based ceramic composites.

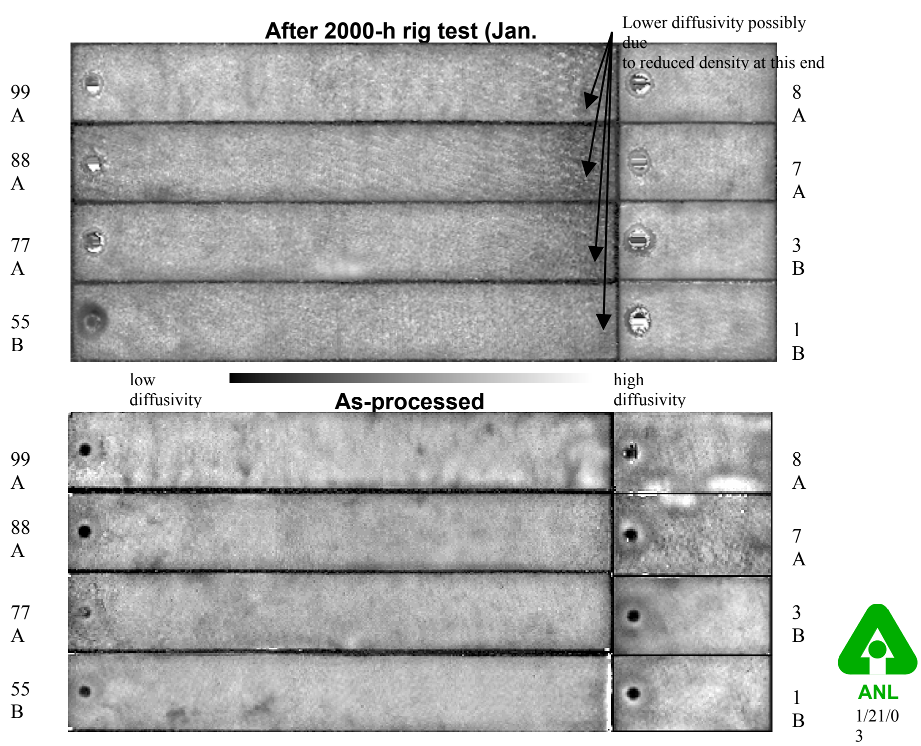


Figure 7. Thermal diffusivity images of oxide/oxide CMC after 2000 hours

NDE Technology for EBCs

This period, we began our interaction with Northwestern University as the first new EBC material was delivered. We are in the process of establishing the optical transmission characteristics and will report on these next period.

Status of milestones

Complete evaluation/demonstration of data acquisition with new large-area X-ray detector. (March 2003)

Complete laser scatter NDE of EBC system from Northwestern University. (April 2003)

Industry/National Lab Interactions

Many new discussions took place this period that is the start of new interactions. These included:

1. Discussions were held with Professor Kathy Faber of Northwestern University relative to new EBC developments for AS800 Si3N4 materials.
2. Discussions were held with Dr. Bjoern Schenk of Honeywell Engines and Systems relative to the EBC development for AS800 and AS950 materials.
3. Discussions were held with Dr. Beth Armstrong of Oak Ridge in regard to EBC developments.
4. Discussions were held with staff at Oak Ridge regarding the transfer of a 420 KVp X-ray head that is necessary in our work for large diameter ceramic components.
5. Discussions were held with staff at COI-ceramics in San Diego relative to the work on oxide-based ceramic composites.
6. Discussions were also held with Ingersoll Rand Energy Systems.

Problems encountered

1. We continue to have a bad array in the RID1640 large area X-ray detector. Until the problem with the large-area CsI detector is resolved, we must continue to use the RID1640. This reduces the data acquisition portion to only the top half of the detector.

Trips/meetings

Trips taken

W. A. Ellingson participated in the Workshop on "'Materials and Practices to Improve resistance to Fuel derived Environmental damage in land-and Sea-based Turbines" held October 22-24, 2002, in Golden, CO.

W. A. Ellingson participated in the Workshop on "Environmental Barrier Coatings" held in Nashville, TN, November 6-7, 2002.

Planned

W.A. Ellingson plans to participate in the 27th Annual International conference on Advanced ceramics and composites to be held January 20-24, 2003, in Cocoa beach, FL.

W. A. Ellingson plans to participate in the DARPA-sponsored MURI-review meeting on thermal barrier coatings to be held January 6-9, 2003, in Santa Barbara, CA.

CHARACTERIZATION OF ADVANCED CERAMICS FOR INDUSTRIAL GAS TURBINE/ MICROTURBINE APPLICATIONS

Oxidation/Corrosion Characterization of Monolithic Si_3N_4 and EBCs

K. L. More and P. F. Tortorelli
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831-6064
Phone: (865) 574-7788, E-mail: morekl1@ornl.gov

Objective

Characterization and corrosion analyses of Si_3N_4 materials provided to ORNL as part of the Hot-Section Materials/Component Development Program.

Exposures of candidate Si_3N_4 materials to high water-vapor pressures (in Keiser Rig) to simulate high-temperature, high-pressure environmental effects associated with microturbines.

Evaluate the reliability of environmental barrier coatings (EBCs) on silicon nitrides for selected microturbine applications.

Highlights

During this reporting period, a study of the microstructural stability of oxide/oxide composites in high water-vapor pressure environments was continued. In this work, the composite material will be subjected to long-term exposures (up to 3000 h) at 1135°C, 10 atm total pressure in a N_2-10%O_2-10%H_2O-6%CO_2 gas mixture. Extensive microstructural and mechanical characterization will be conducted following 0, 1000, 2000, and 3000 h exposures. Composite samples are currently being exposed for the second 1000 h cycle to accumulate 2000 h total exposure on this set of samples.

Technical Progress

A furnace system that provides a high-temperature, high-pressure, low-flow-velocity (< 0.1 fps) mixed-gas environment (ORNL's Keiser Rig) is being used to conduct first-stage evaluations of COI Ceramic's (oxide fiber)/(oxide matrix) ceramic composite material. The material is being evaluated for Capstone Turbines in support of their Advanced Microturbine Systems Program. The oxide/oxide composite will be subjected to long-term exposures (0-3000 h) in a simulated microturbine environment; 1135°C, 10 atm total system pressure, and a N_2-10%O_2-10%H_2O-6%CO_2 gas mixture.

A total of 36 specimens were submitted to ORNL for evaluation and exposure. Eighteen 1” X 7” bars (to be used primarily for pre- and post-exposure tensile tests) and eighteen 1” X 2” coupons (to be used for pre- and post-exposure microstructural characterization and shear tests) were received in July 2002. Each coupon was screened by NDE (using digital radiography and thermal diffusivity techniques) at Argonne National Laboratory (ANL) prior to sending to ORNL. Four specimens of each type (8 total) will be used for the microstructural and mechanical evaluation in the as-processed condition (0 h) as well as following 1000, 2000, and 3000 h exposures in the Keiser Rig. After each exposure and prior to post-test characterization at ORNL, the exposed coupons will be returned to ANL for post-test NDE for comparison with NDE results obtained for each coupon before exposure in the Keiser Rig.

The first (1000 h) exposure of the COI Ceramics oxide/oxide composite material began in mid-August, 2002 and ended November 14, 2002. At that time, all 24 exposed specimens were removed from the Keiser Rig, 8 specimens were selected and removed for the 1000 h NDE, microstructural, and mechanical characterization. The remaining 16 samples were put back in the Keiser Rig (in the exact same positions in tube) for another 1000 h exposure (to accumulate 2000 h total on second set of samples). The second 1000 h exposure began on November 21, 2002 and is scheduled to end on January 12, 2003 at which time another 8 samples exposed for 2000 h will be removed from the test for NDE followed by microstructural and mechanical evaluation.

In addition to Keiser Rig exposures, rectangular (~2.5 x 1 cm) coupons of the oxide/oxide composite were exposed for 500 h at 1135°C at 1 atm in flowing dry air or air + 10 vol% H_2O in a microbalance so that stability over an extended period of time could be monitored gravimetrically. In both environments, a very large mass loss (on the order of 1-2%) occurred during heating (~10 min). Subsequently, a slow, but significant, mass gain was recorded over the exposure period, thereby indicating formation of some type of reaction (oxidation) product. In the case of the air + 10%H_2O exposure, the rate of mass gain was greater than when the composite was exposed to dry air. After the initial mass losses, the mass of the coupon exposed to air + 10%H_2O increased ~0.20% over 500 h compared to ~0.05% for the coupon held in dry air.

Status of Milestones

08/2001 Complete 2000 h exposures on three different Honeywell Si_3N_4 materials in ORNL’s Keiser Rig, characterize microstructural changes, and determine material recession rates. Report results.
Milestone is completed. Results were reported/presented at Honeywell Engines & Systems on March 20, 2002.

07/2002 Complete evaluation of uncoated Si_3N_4 exposed to simulated microturbine operating conditions in the Keiser Rig for three temperatures and two water-vapor pressures and report results.
Milestone will only be partially completed since original Si_3N_4 material for this program, manufactured by Honeywell Ceramic Components, will no longer be evaluated in the Keiser Rig. To date, exposures at one temperature (2400°F) and two water-vapor pressures (0.3 atm and 2.0 atm) have been completed with ~1000 h accumulated at one additional temperature (2200°F).

08/03 Report results of initial evaluation of "new" Si_3N_4 materials from Saint-Gobain and Kennametal exposed in the Keiser Rig for two temperatures and two water-vapor pressures.
New Si_3N_4 materials have not yet been received from either St. Gobain or Kennametal.

Industry Interactions

Attended "Environmental Barrier Coatings Workshop" in Nashville, TN on November 6-7, 2002. Met with several Dave Carruthers to discuss collaborative work on Keiser Rig exposures of oxide/oxide composites and characterization at Oak Ridge National Laboratory.

Visited GE Aircraft Engines, Cincinnati, OH on November 24, 2002 to discuss BSAS-based EBC characterization work being conducted at ORNL.

Problems Encountered

None

Publications/Presentations

K. L. More and P. F. Tortorelli, "Evaluation of EBCs in ORNL's Keiser Rig," presented at the EBC Workshop in Nashville, TN, November 6-7, 2002.

Mechanical Characterization of Monolithic Silicon Nitride Si_3N_4

R. R. Wills, M. Pierson, S. Hilton, and S. Goodrich
University of Dayton Research Institute
300 College Park, KL-165, Dayton, OH 45469-0172
Phone: (937) 229-4341, E-mail: roger.wills@udri.udayton.edu

Objective

The objective of this project is to work closely with microturbine materials suppliers to characterize monolithic ceramics and provide the data obtained to microturbine manufacturers via the website database as well as user-friendly software, which will allow prospective users to readily compare different silicon nitrides. This project consists of the following four tasks:

- Task 1: Evaluate Strength and Slow Crack Growth of New Materials
- Task 2: Modify Six Existing Creep Frames to Allow Introduction of Water Vapor
- Task 3: Evaluate the Effects of Water Vapor Upon Honeywell's Silicon Nitride Ceramics
- Task 4: Develop "User Friendly" Software for Searching Existing Mechanical Properties Database

Task 1 is motivated by material needs of Ingersoll-Rand (IR) Energy Systems, General Electric, and UTRC. The ceramic materials being considered by IR Energy Systems include Kyocera's SN235 and SN237 for which the required mechanical property data are somewhat limited. In the case of the UTRC microturbine, Si-SiC is a prime candidate for the combustor. The goal of Task 2 is to modify existing facilities to evaluate the effects of water vapor upon the strength and creep rupture behavior of both uncoated and coated Si_3N_4 ceramics. In Task 3, the facilities developed in Task 2 will be used to evaluate the mechanical behavior of specimens with and without an environmental barrier coating (EBC).

Highlights

A potential EBC ceramic has been identified. This material, an aluminum silicon carbide,Al_4SiC_4, is isostructural with alpha silicon carbide. While literature data suggests it has excellent resistance to oxidation little is known about the properties of this material.

The web site has been modified to include a database for designers that include the individual data points for fast fracture and creep rupture. The data is in the form of an Excel spreadsheet with thirteen worksheets containing the data for various silicon nitrides and carbides.

Technical Progress

Upgrading the Website

The "Structural Ceramics Property Database" on the UDRI website (http://www.udri.udayton.edu/handbook/TOC.html) was set up several years ago and we are currently re-evaluating its appearance, content and use with the help of Dr. Mattison Ferber at ORNL.

General Electric suggested that it would be useful to have all the individual datapoints from the University of Dayton Research Institute's(UDRI's) strength measurements available for their design studies. UDRI has been constructing this database in the form of an EXCEL spreadsheet with a separate worksheet for each material. The database contains tensile and flexural strength as well as creep rupture for all the silicon nitrides and carbides tested to date. Data for only longitudinally ground samples is included. Since the intent of this database is for design purposes only data pertaining to fast fracture and creep studies are included. The excel spreadsheet contains worksheets for the following materials:

Sintered Hexalloy SA silicon carbide
Liquid Phase sintered silicon carbide
Coorstek SC2 reaction bonded silicon carbide
Norton NC230 reaction bonded silicon carbide
Norton NT 154 silicon nitride
Norton 164 silicon nitride
Kyocera SN237,SN235,SN253,SN 88A,SN282 silicon nitrides
Ceradyne 146 hot pressed silicon nitride
Honeywell AS800 silicon nitride

Aluminum Silicon Carbide-A Potential EBC

Aluminum silicon carbide Al_4SiC_4 forms a solid solution with alpha silicon carbide[1]. The high aluminum content in this ceramic suggests it may have good oxidation resistance, and thus could be a candidate EBC. While little is known about this ceramic Itatani et al [2] heated it in air at 1500 C for 1 hour. X ray diffraction indicated the presence of Al_2O_3,$3Al_2O_3.2SiO_2$ and Al_4SiC_4 suggesting a thin reaction layer, which they estimated as 25 microns in depth with Al_2O_3 as the outer layer. This could be a very protective layer.

Status of Milestone

(a) Complete modification of six creep rupture frames to accommodate testing in water vapor and complete validation tests (due August 1, 2001/completed).

(b) Complete test matrix for Honeywell Ceramic Components and issue letter report (due September 2002/off schedule). No samples have been received yet. This milestone will have to be modified given the uncertainty concerning the availability of the Honeywell material.(This milestone has been removed in the new Statement of Work)

Industry Interactions

Roger Wills attended a DOE workshop hosted by the South Carolina Institute for Energy Studies for the National Energy Technology Laboratory. The meeting was held in October at the University of Connecticut. The major turbine engine manufacturers presented their interests and needs and existing University contractors presented their results to date on their programs. The meeting concluded with a panel discussion.

On November 6th and 7th Roger Wills attended and presented a. paper at the microturbine Environmental Barrier Coatings Workshop held in Nashville. The paper was entitled "Potential EBCs from the R-Si-Al-O-C-N System and Test techniques for Microturbine Ceramics". The paper made recommendations for several possible barrier materials, and how to evaluate candidate materials. In addition some new laboratory tests were proposed to enable better assessment of candidate EBCs prior to their evaluation in engine tests. These tests would enable time dependent stress, strain and corrosion data to be obtained as well as evaluating coatings on tubes.

Dr Reza Saraffi-Nour and Curtis Johnson of General Electric requested UDRI to conduct some Chevron testing to determine fracture toughness. They would also like the strain measured during the test. Dr Mattison Ferber sent us some long silicon carbide extensometer rods to enable us to perform the strain measurements. The samples have not yet been received from General Electric.

Problems Encountered

None

Publications/Presentations

A paper entitled "Potential EBCs from the R-Si-Al-O-C-N System and Test techniques for Microturbine Ceramics" was given at the Environmental Barrier Coatings Workshop in Nashville, Tennessee.

Reference

1. J. Schoennahl, B. Miller and M. Daire in Materials Science Monographs 4:Sintering-New Developments-Elsevier Science Publishing, Amsterdam,1979.

2. K. Itanani, F. Takahashi, M. Aizawa, I. Okada,I.J.Davies , H. Suemasu and A. Nozue, "Densification and microstructural developments during the sintering of aluminum silicon carbide," J. Maters. Sci. 37,335-342, 2002.

Hot Section Components in Advanced Microturbines

Bjoern Schenk, Honeywell Engines, Systems & Services (HES&S)
D. Newson, J. Nick and J. Wimmer, Honeywell Ceramic Components (HCC)
Chien-Wei Li, Honeywell Laboratory (HL)
Honeywell Engines, Systems & Services
2739 E. Washington Street
P.O. Box 5227, Phoenix, AZ 85010
Phone: (602) 231-4130, E-mail: bjoern.schenk@honeywell.com

Objectives

- Determine the corrosion resistance of AS800 and AS950 silicon nitrides in high water vapor environments representative of microturbine engines.
- Demonstrate the effectiveness of environmental barrier coatings to protect silicon nitride materials from corrosion/material recession in microturbine environments.
- Refine processes to manufacture gelcast AS950EXP material, for use in turbine engine applications.

Highlights

- Task 4 was completed. All Keiser rig specimens have been shipped to Honeywell Labs for EBC development activities.
- Mechanical properties generated from gelcast AS950EXP were unacceptable, further material / process developments are required.
- Sandia completed the finite element thermal model of the large ABB HIP furnace at HCC.

Technical Progress

Phase II, Task 4 – AS800 and AS950EXP specimen preparation for EBC development

Slipcast AS800 Keiser rig specimens (75 as-processed specimens and 25 ground specimens) were fabricated and shipped to Honeywell Labs for EBC development activities. This completed Task 4. The next generation EBC development activities using these specimens are conducted under an internally funded Honeywell R&D program.

Phase II, Task 5 – AS950EXP Gelcast Substrate Process Refinement

Dimensional inspection of the AS950EXP generic nozzle ring, reported on last quarter and shown in Figure 1, was completed. Roundness and true position measurements indicated that the AS950EXP part fell within the same population as AS800 nozzle rings.

Mechanical property data was generated for gelcast AS950EXP material, processed under the revised heat treatment process, reported on last quarter. The achievement of 99.6% density was a milestone for gelcast AS950EXP, however, neither the room temperature strength nor the dynamic fatigue properties at 1371ºC were as good as current AS800. This degradation of the properties is attributed to a change in secondary phase composition due to the revised thermal treatment.

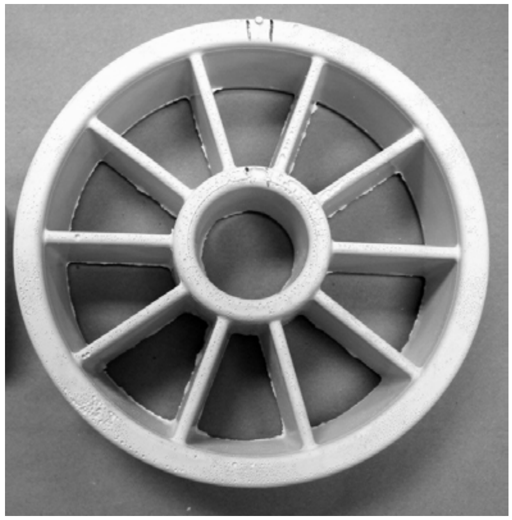


Figure 1. AS950EXP gelcast generic nozzle ring made with new – improved initiator system.

After binder burnout furnace upgrades had been completed, material was processed using a modified cycle. Carbon analysis of the material was performed and found to be uniform in plates located throughout the oven.

A finite element thermal model of the furnace used to densify AS950EXP has been created at Sandia National Laboratory. This model duplicates the furnace geometry and materials of construction including the Carbon/Carbon heating elements. The model also simulates the crucibles used in sintering along with a typical payload. In addition, the model simulates the thermocouples and proportionate control system used to control both upper and lower zones of the furnace. A major unknown input to the model was the thermal characteristics of the furnace insulation, also referred to as the thermal barrier. As a result, the properties of the barrier were varied until the model accurately predicted the previously measured cool-down curve for the furnace. The model is currently being run on Sandia National Laboratory's Janus machine using 200 parallel processors. Figure 2 shows the results of a test case run.

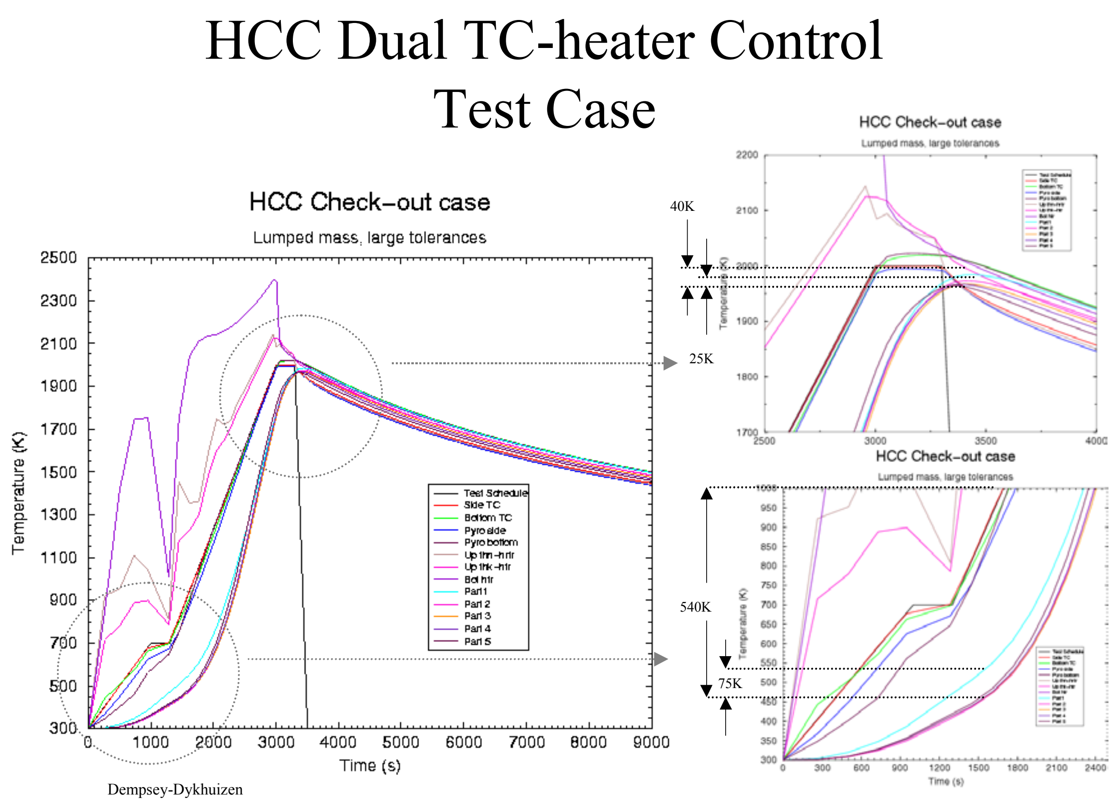


Figure 2. Finite element thermal model output for large ABB furnace at HCC

Outputs of the model include side and bottom thermocouple temperatures, side and bottom pyrometer target temperatures, furnace heating element temperatures, and the temperature of five parts, distributed from top to bottom of the furnace.

In general, the model correctly predicts the observed furnace behavior. However, in order to calibrate the model, additional temperature measurements are required at various locations in the furnace during a typical sintering cycle. Instrumentation of the furnace is planned for early 2003. Once good agreement between the observed and predicted temperatures is obtained, the model can be used to predict temperatures and temperature variations throughout the furnace as well as evaluate changes to the sintering cycle heating rates and hold times. The goal is to minimize thermal gradients in the furnace while ensuring that the parts being sintered experience the desired temperature schedule.

Status of Milestones

No milestones were due during this reporting period.

Industry Interactions

Honeywell's Ceramic Program Director, Dr. Bjoern Schenk, initiated an R&D collaboration with the University of Colorado at Boulder on the development of next generation EBC material systems. Discussions were held with various small businesses regarding the joint development of PDC-based EBC systems. Contract negotiations with a potential EBC development partner in Europe are progressing as planned.

Problems Encountered

None

Publications/Presentations

Honeywell's Ceramic Program Director, Dr. Bjoern Schenk, attended the ORNL-organized EBC Workshop in Nashville, TN, November 6-7 and presented Honeywell's progress and plans for the Advanced Microturbine Systems program, and status updates on the Hot Section Components for Advanced Microturbines Program, the High-Velocity High Pressure Burner Rig (HSBR) Program, as well as EBC development activities.

Microstructural Characterization of CFCCs and Protective Coatings

K. L. More and P. F. Tortorelli
Metals and Ceramics Division
Oak Ridge National Laboratory
P.O. Box, Oak Ridge, Tennessee 37831-6064
Phone: (865) 574-7788, E-mail: koz@ornl.gov

Objective

Characterization of CFCC materials and CFCC combustor liners after exposure to simulated (ORNL's Keiser Rig) and actual (engine tests) combustion environments

Exposures of candidate environmental barrier coatings (EBCs) to high water-vapor pressures (in Keiser Rig) to determine thermal stability and protective ability

Work with CFCC and coating suppliers/manufacturers to evaluate new/improved ceramic fibers, protective coatings, and composite materials

Highlights

During this quarter, the microstructural and mechanical characterization of a set of engine-tested CFCC liners removed after 15,144 h from a Solar Turbines Centaur 50S SoLoNOx gas turbine (Malden Mills co-generation test site in Malden, MA) was started.

Technical Progress

As reported previously (DER Quarterly Report for July 1, 2002 – September 30, 2002), the inner and outer EBC/CFCC combustor liners were removed from the Centaur 50S engine at Malden Mills on July 8, 2002 after 15,144 h field-testing and 92 starts. The inner liner was a Tyranno fiber-reinforced MI-processed liner made by Goodrich Corp. and the outer liner was a Hi-Nicalon –reinforced CVI liner produced by GE Power Systems Composites. Each liner had a SiC seal coat and a plasma sprayed Si/BSAS+Mullite (mixed)/BSAS EBC applied by UTRC. NDE was conducted at Argonne National Laboratory (ANL), after which the liners were returned to Solar Turbines.

A detailed sectioning plan was established by ORNL (with input from the other program participants) for both the inner and outer liners such that all the program participants received sections of interest from the two liners. Two or three pieces were cut from each liner and were sent to ANL, UTRC, Goodrich, and Solar Turbines, Inc., for their own internal characterization and evaluation. ORNL retained a majority of the sections from both liners since ORNL led the microstructural and mechanical characterization effort.

As shown in Figure 1, which shows the correlation between the NDE (thermal diffusivity data) and actual liner gas-path surface, several areas of the inner liner were sectioned for detailed microstructural evaluation (#1-1, #1-5, #2-1, #4-1, and #6-5 as shown by green boxes). These sections were chosen specifically to fully characterize several observations made both visually and by NDE, including a large sub-surface defect and EBC recession (section #1-1 and #2-1), crack formation (section #1-5 and #4-1), and extensive EBC recession associated with fuel injector impingement (section #6-5).

Section #1-1 provided a significant amount of information regarding the inner liner and possible life-limiting materials degradation mechanisms. The sub-surface defect identified by NDE (which was present in the as-processed liner) was observed in the cross-section microstructural images of inner liner section #1-1. The sub-surface composite oxidation/damage associated with this defect (presumably a de-lamination between fiber layers that occurred during initial composite densification) is shown in Figure 2. Sub-surface composite oxidation occurred around the defect during engine testing. SiC/SiC composite oxidation rates are more rapid than rates measured for monolithic SiC at elevated water-vapor pressures, thus, oxidation-induced damage will accumulate much more rapidly within the composite material.

Not only was extensive oxidation observed around the pre-existing sub-surface defect within the composite material, but a much greater amount of oxidation occurred from the back (non gas-path) surface of the inner liner, as shown in Figure 3. In fact, non gas-path surface recession was observed around the entire circumference of the inner liner. The composite experienced a significant loss of thickness in many areas from the liner non-combustion side. There were several reasons for the extensive non gas-path surface oxidation: (1) no EBC was applied to this surface – previous engine tests have shown that the non-combustion surface does not experience significant oxidation due to somewhat lower temperatures, much lower water-vapor pressures, and lower gas velocities, thus, no EBC is used for the protection of this surface, (2) the thickness of the CVD SiC seal coat was much thinner than expected – previous liners prepared for engine testing have had seal coats measuring 300-450 μm in thickness, however, the MI inner liner used for the Malden Mills engine test had a SiC seal coat thickness of only ~100 μm, and (3) large cracks formed during the early stages of engine testing (<5000 h) - the engine test was continued for many more hours after the cracks formed, thus, oxidation around the cracks could have easily initiated extensive oxidation of the non gas-path surface.

The microstructure of the as-processed BSAS EBC top-coat sprayed on the inner liner was not optimum. As shown in Figure 4, the BSAS top-coat (image was taken from the cool aft end of the liner which should be representative of the as-processed microstructure) was not fully dense and appeared quite friable. Even though the EBC had so many processing defects (primarily large-scale porosity), nearly all of the EBC remained intact during engine testing (as evidenced by the photograph of the inner liner gas-path surface shown in Figure 1). However, the EBC on the inner liner did experience measurable recession within the center portion of the liner, especially in areas associated with fuel injector impingement. Section #6-5 (see Figure 1) clearly showed a fairly large fuel injector impingement area where EBC recession was severe; full recession of the EBC occurred such that the underlying CFCC began to oxidize. These observations were consistent with results presented previously for EBC/CFCC engine tested liners.[1]

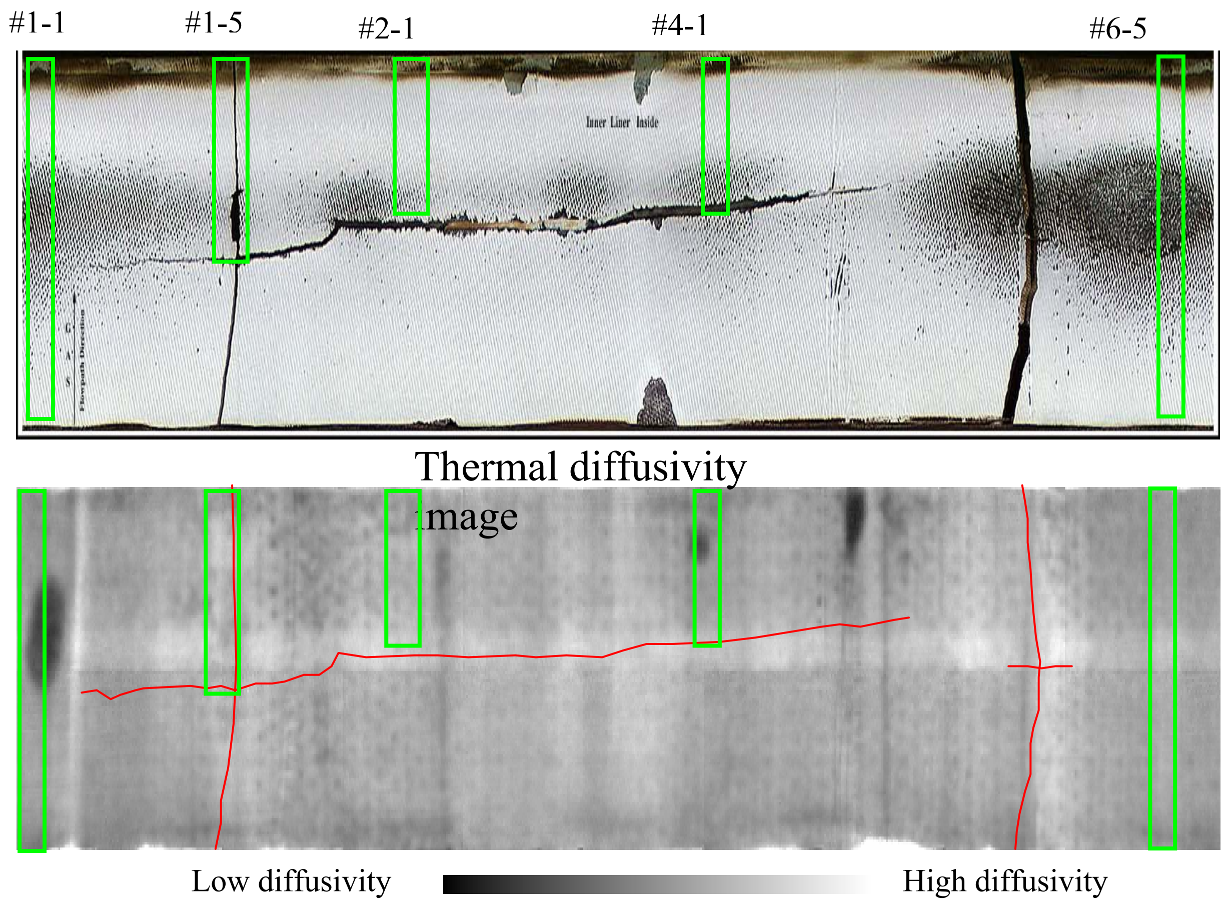


Figure 1. Photograph of inner liner gas-path surface after 15,144 h engine test at Malden Mills with the associated thermal diffusivity image. The red lines indicate the position of cracks in the liner on the thermal diffusivity image. The green boxes show where sections were taken for microstructural analysis.

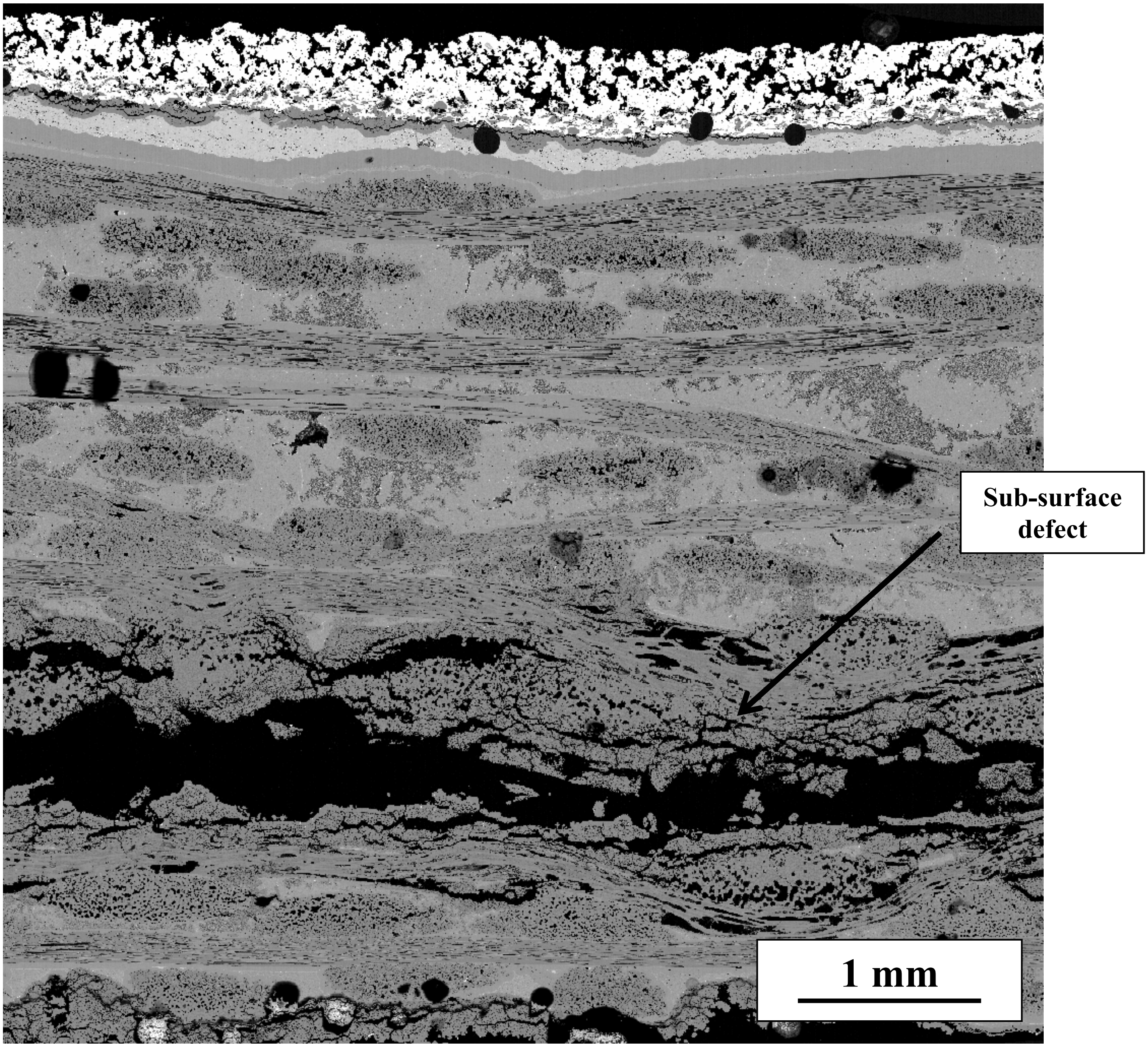


Figure 2. Sub-surface defect initially identified by NDE. Extensive oxidation around the defect edges (and early NDE data) suggests that the defect was present as a de-lamination in the as-processed liner.

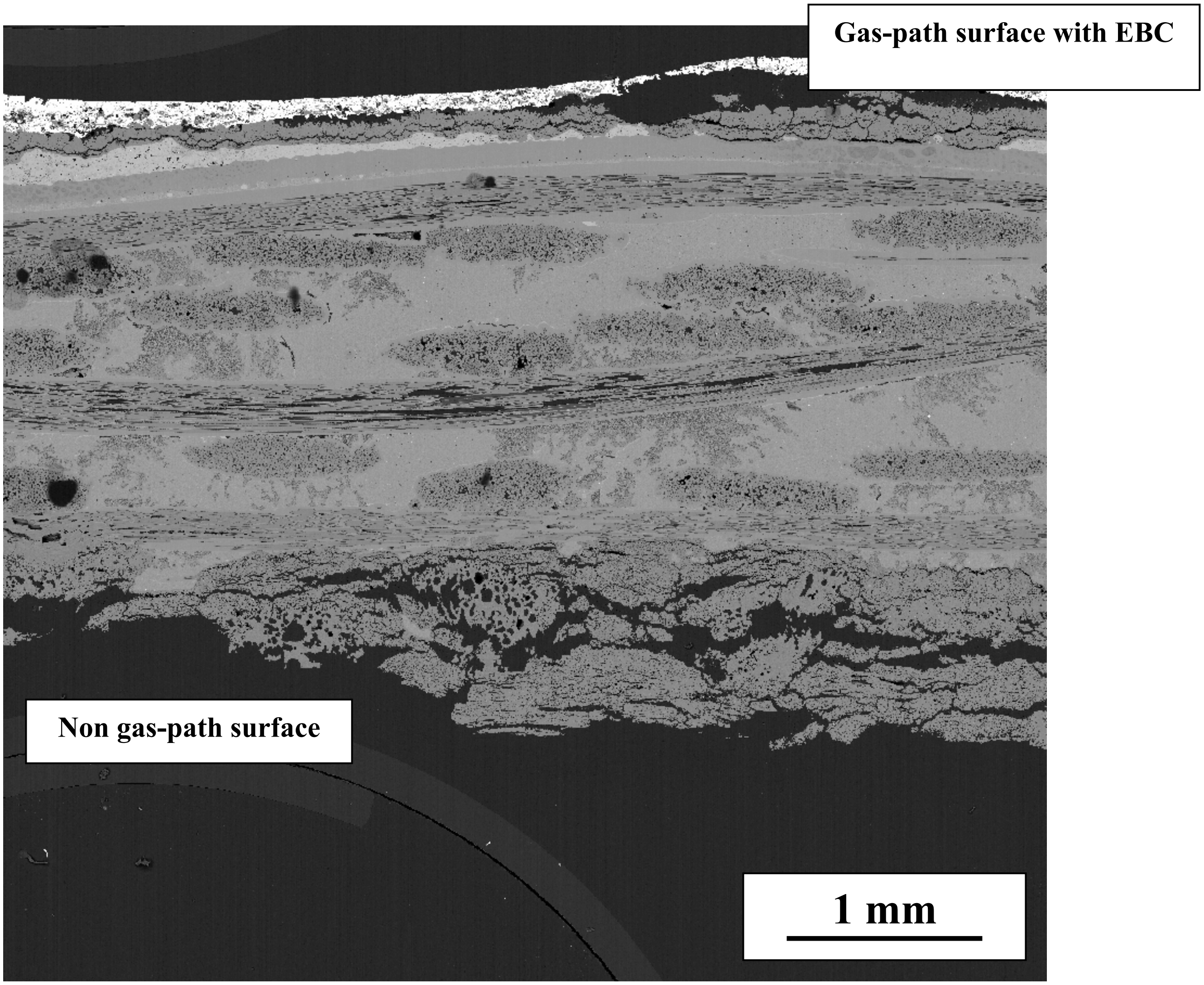


Figure 3. Significant CFCC recession was observed on non-gas-path surface of inner liner. Note that EBC is still present on gas-path surface and all thickness loss occurred from the back side of liner.

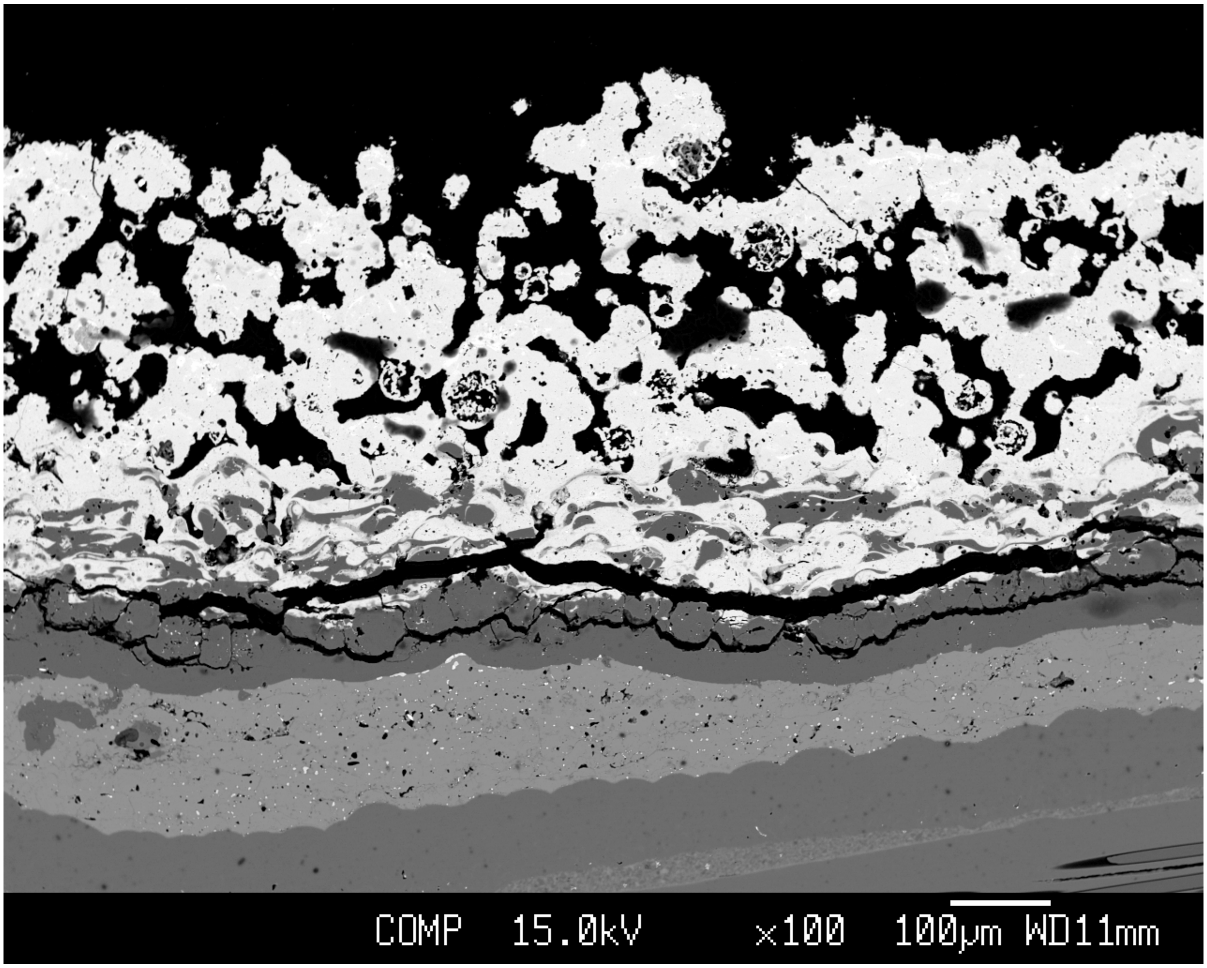


Figure 4. Microstructure of EBC (from cool aft end of inner liner) showing non-optimized BSAS top-coat with large-scale open porosity.

Status of Milestones

03/02 Milestone — Complete a comprehensive report on oxidative degradation mechanisms of Si-based ceramics and rates of such materials at high temperatures and high water-vapor pressures typical of gas turbines.

This milestone has been completed. P.F. Tortorelli and K.L. More, "Effects of High Water-Vapor Pressure on Oxidation of SiC at High Temperature," paper was submitted for publication in *Journal of the American Ceramic Society*.

04/02 Milestone — Prepare a report and present results on the evaluation of the set of 14,000h Texaco (Chevron) combustor liners.

This milestone has been completed. A manuscript has been published by ASME and is part of the proceedings of the IGTI Turbo Expo 2002 in Amsterdam, June 3-6, 2002. K.L. More, P.F. Tortorelli, L.R. Walker, J.B. Kimmel, N. Miriyala, J.R. Price, H.E. Eaton, E.Y. Sun, and G.D. Linsey, "Evaluating EBCs on Ceramic Matrix Composites After Engine and Laboratory Exposures," ASME Paper #GT-2002-30630.

ASME Paper #GT-2002-30630 was awarded 2002 Best Paper by the Ceramics Committee of IGTI as well as being selected for publication in Journal of Engineering for Gas Turbines and Power.

04/03 Milestone — Prepare a report and present results on the evaluation of the set of 15,000 h Malden Mills engine-tested combustor liners.

Work is in progress.

09/03 Milestone — Complete first-stage evaluation of the effects of water vapor on the oxidation of at least two new candidate compositions for protective coatings.

Work is in progress.

Industry Interactions

Attended "Environmental Barrier Coatings Workshop" in Nashville, TN on November 6-7, 2002. Met with several industrial contacts (including UTRC and COI Ceramics) to discuss collaborative work on Keiser Rig exposures and characterization at Oak Ridge National Laboratory.

Problems Encountered

None

Publications/Presentations

1. K. L. More and P. F. Tortorelli, "Evaluation of EBCs in ORNL's Keiser Rig," presented at the EBC Workshop in Nashville, TN, November 6-7, 2002.
2. K. L. More, P. F. Tortorelli, L. R. Walker, H.E. Eaton, E. Y. Sun, G. D. Linsey, J. B. Kimmel, N. Miriyala, and J. R. Price, "Evaluating EBCs on Ceramic Matrix Composites After Engine and Laboratory Exposures," presented at IGTI Turbo Expo 2002, Amsterdam, June 3-6, 2002. ASME Paper #GT-2002-30630. Selected for publication in *Journal Engineering for Gas Turbines and Power*.
3. K.L. More and P.F. Tortorelli, "The High-Temperature Stability of SiC-Based Composites in High Water-Vapor Pressure Environments," submitted for publication in *Journal of The American Ceramic Society*.
4. P.F. Tortorelli and K.L. More, "Effects of High Water-Vapor Pressure on Oxidation of SiC at High Temperature," submitted for publication in *Journal of The American Ceramic Society*.

References

1. K.L. More, P.F. Tortorelli, L.R. Walker, H.E. Eaton, E.Y. Sun, G.D. Linsey, J.B. Kimmel, N. Miriyala, and J.R. Price, "Evaluating EBCs on Ceramic Matrix Composites After Engine and Laboratory Exposures" ASME Paper #GT-2002-30630.

High Speed Burner Rig Development

Bjoern Schenk and Glen Schroering
Honeywell Engines, Systems & Services
2729 E. Washington Street, P.O. Box 5227
Phoenix, AZ 85010
Phone: (602) 231-4130, E-mail: bjoern.schenk@honeywell.com

Objectives

Design, build, and operate a burner rig facility which

- will provide ability to expose most promising ceramics and coatings at environmental conditions typical of turbine nozzles and blades
- will provide oxidation information at conditions well beyond current experimental database

Test section maximum operating conditions

- Gas Temperature - 3000°F
- Average Gas Velocity - 2700 ft/s
- Partial Pressure of Water Vapor - 70 psia (in combustor)
- Stress Rupture Test Capability
- Durability for extended unmanned operation (100's of hours)
- Operating costs to be minimized

Highlights

- INSTRON load test machine refurbished – able to control motion both up and down at variable speeds.
- External energy sources for the facility have been staged.
- Initial test plan written.

Technical Progress

A thermal/stress analysis was conducted on the latest combustor module (combustor can, liner and outer housing) configuration. The stress levels were acceptable and the selected vendors were allowed to begin fabrication.

Honeywell is working with Williamson Corporation to incorporate two of their optical pyrometers in the test section. Focal length and spot size are the major issues in this application.

The INSTRON load test frame was re-wired. Motion in both directions at variable speeds was demonstrated. The remaining task is to interface it with a suitable control package.

Status of Milestones

No milestones were due during this reporting period

Industry Interactions

None

Problems Encountered

A 3-D analysis of the test section revealed high stresses at the three locations where 1/8" holes were to be machined for optical temperature measurements. Sapphire windows were to be placed at those locations. The ANSYS model will be reviewed and possibly refined. Fabrication of the test section is on hold until this is resolved.

Publications/Presentations

Honeywell's ceramic program director, Dr. Bjoern Schenk, attend the ORNL-organized EBC Workshop in Nashville, TN, November 6-7 and presented Honeywell's progress and plans for the Advanced Microturbine Systems program and status updates on the Hot Section Components for Advanced Microturbines Program, the High-Velocity High Pressure Burner Rig (HSBR) Program, as well as EBC development activities.